AMRA CR 64-04/3

Control of Cast Grain Size of Steel Castings-Effect of Structure and Nonmetallics on Properties

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by

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January, 1968

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U. S. ARMY MATERIALS RESEARCH AGENCY
WATERTOWN, MASSACHUSETTS 02172

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U.S. ARMY MATERIALS RESEARCH AGENCY

WATERTOWN, MASSACHUSETTS 02172

ABSTRACT

This project was concerned with determining the influence of micro-structural refinement on the mechanical properties of cast high strength 4335 steel. The strength levels investigated were approximately 250,000 psi tensile strength and 205,000 psi yield strength obtained with a liquid quench and tempering treatment. The steel was cast in unidirectionally solidified cylinders and conventional sand mold keel blocks.

It was determined that the addition of approximately 0.10% Ti to the steel subsequent to deoxidation provided considerable structural refinement. This structural refinement consists of changing the usual columnar structure to equiaxed and of refining the spacing of the secondary dendrite arms. Such refinement was accompanied by improvements in the tensile ductility and impact resistance of the steel. This improvement in properties was particularly marked on specimens located with their long axis perpendicular to the direction of heat flow. Structural refinement improved both the level of mechanical properties and reduced their directionality.

Studies of the micro-structure of the cast steel determined that nonmetallic inclusions were the primary factor (in the absence of microshrinkage) in determining the level of ductility. The loss in ductility that occurred because of the presence of these nonmetallic inclusions was reduced by low sulfur contents and by means of combined deoxidation treatments. The best deoxidation treatments were found to be calcium-manganese-silicon or zircon-silicon added together with aluminum. Improved ductility was also obtained with the final secondary dendrite arm spacing that is obtained by rapid solidification rates and by grain refinement with titanium.

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INTRODUCTION

The control of the solidification process is a basic step in obtaining the potential mechanical properties of a casting. The as-cast structure and presence of heterogeneities such as porosity and nonmetallic inclusions or nonequilibrium segregates determine the degree to which the properties inherent in the casting are realized. The type and fineness of the as-cast structure is determined by the composition, solidification time and the state of nucleation during solidification. The presence of heterogeneities in steel is primarily governed by the thermal gradient in the liquid during solidification, the chemical composition and the deoxidation practice.

Grain refinement is one method to control the solidification process. Nonferrous alloys are frequently grain refined to achieve better strength, ductility, resistance to cracking and hot shortness. Similarly, cast irons are frequently grain refined to control the structure and its properties. A detailed review of this subject was presented in earlier reports. Grain refinement of steel is commercially applied only in some industries. The application of a moving electromagnetic field during solidification has been reported to refine the structure of continuously cast strands and to improve the surface condition, reduce macroporosity and the tendency for hot tearing. Macrorefinement (conversion of a columnar dendritic structure to an equiaxed structure) has been obtained in some instances by applying a particular melting, deoxidation or inoculation practice. In spite of these reports, grain refinement has not assumed the important role in the steel foundry industry which it has achieved in the nonferrous or cast iron industry.

Recently, a technique of grain refining high strength, low alloy steel of the type AISI 4335 was developed. This technique consists of inoculating the melt with titanium. A suitable titaiuum inoculation reduces the dendrite spacing of the casting and produces an equiaxed dendritic structure in a casting, which would normally solidify in a columnar dendritic manner. Such refinement improves the tensile ductility as measured by the reduction in area and toughness measured by Charpy V-notch impact specimens.

The present investigation was undertaken to achieve the following objectives: 1) improvement of the degree of refinement without introducing embrittling, nonmetallic inclusions; 2) establishment of the relation between wall thickness, or equivalently solidification time and structure of properties; 3) influence of melting variables such as deoxidation practice on the structure, inclusions and properties; 4) determination of the influence of the mode of solidification (equiaxed versus columnar dendritic) on the segregation behavior of solute elements and on the incidence of microporosity.

PROCEDURE AND MATERIALS

The alloy used in this investigation is a high strength, low alloy steel of AISI 4335 type with the following nominal composition:

C%	Mn%	Si%	Cr%	Mo%	Ni%
0.30 to 0.35	0.60 to 0.80	0.20 to 0.35	0.70 to 0.90	0.20 to 0.30	1.65 to 2.00

The steel was generally melted at a sulfur level of 0.013 percent with 0.01 percent phosphorus. Exceptions to this analysis will be referred to in the text whenever necessary. The steel was melted from closely controlled charge materials using 50 and 100 pound, magnesia lined, high frequency induction furnaces. The primary charge of low sulfur iron was rapidly melted and a CO boil initiated and maintained for at least two minutes by adding pig iron. Pig iron was used to maintain the boil and to adjust the carbon content after blocking the heat with ferrosilicon and ferromanganese. Standard ferroalloys and electrolytic nickel were employed to introduce the necessary alloying elements. A protective atmosphere was maintained during the preparation of the heat by directing a stream of argon at the melt through a refractory cover. Final deoxidation was performed by adding 0.10 percent aluminum prior to tap. During the phase of the investigation of the effect of deoxidation practice all deoxidizers were wrapped in light gage steel and plunged below the surface of the melt.

Unidirectionally solidified cylindrical castings and standard keel blocks were employed as test castings. The castings were poured directly from the furnace into molds through a pouring dish. The cylindrical castings were used in two sizes weighing 45 and 11 pounds. These castings will be referred to as the large and small cylindrical casting respectively. The bottom of the mold cavity was formed by a graphite chill block; the rest of the mold cavity was formed by an exothermic sleeve separated from the chill block by a thin dry sand ring. Exothermic hot topping was used for all large cylinders, none was employed for the smaller cylinders. The standard keel block castings with 1" x 1" x 8" long legs were cast in dry sand molds with a small amount of exothermic hot topping.

The castings were usually produced in pairs from a single melt, first the base or uninoculated casting was poured, then the melt was treated and the inoculated casting poured. The ferrotitanium employed for grain refinement was plunged similar to the deoxidizers mentioned earlier. The time between inoculation

and pouring was held uniformly at two or three minutes. The pouring temperature ranged from 2800° to 2950°F for large cylinders as discussed in the text. For small cylinders and keel blocks, a uniform pouring temperature of 2900°F was selected. The pouring temperature was closely controlled by immersion thermocouples.

Macro and microexamination of the structure was conducted in all castings. Humphrey's reagent was used to develop the dendritic structure. The details of the cast structure were improved in some cases by isothermal transformation heat treatment. This consisted of austenitizing and quenching to the isothermal transformation temperature of 1200°F and resulted in ferrite-pearlite dendrites and a martensitic matrix. The primary dendrite and secondary arm spacings were measured at various heights of the cylindrical castings as indicated in the text. The secondary dendrite arm spacing of keel block legs was measured 3/16 inches from the cast surface at 3/4 inches from the bottom of the leg. The amount of nonmetallics was quantitatively determined employing a two-dimensional systematic point count method.

A microanalysis was conducted on specially prepared metallographic specimens employing an electron microprobe, model 400, built by the Materials Analysis Company. Specimens used for microprobe analysis were isothermally transformed to provide maximum detail in the etched structure. When solute maxima were determined, the specimens were isothermally transformed up to 90 minutes in order to transform all of the structure, except the areas exhibiting a solute maximum to ferrite plus pearlite. The material exhibiting a solute maximum transformed to martensite during the following quench in water. The path along which the composition was to be determined was marked by micro hardness impressions. The specimens were then repolished and analyzed. The microprobe traces were revealed by etching the specimens with 3 percent nital.

All microprobe analyses were made by point counting, integrating for ten seconds. The distance between points varied from 2 to 20 microns depending on the accuracy required. In areas exhibiting a solute maximum the distance between points was two to five microns. The microprobe was operated with a potential difference between filament and target of 25 KV, the specimen current was 0.03 microamperes, the take-off angle was 35°, the beam diameter was held between one and two microns. The elements manganese and chromium were analyzed quantitatively using specimens of known composition as standards; qualitative analyses were made for titanium.

The method used to determine solute minima and maxima consisted of conducting point counts along diagonal paths through the dendrite center into the geometrical center between adjacent dendrites of a columnar dendritic specimen. For equiaxed structures, the analysis was restricted to specimens which were slightly over inoculated so that the specimen contained titanium sulfides instead of the usual manganese sulfides. The reason for selecting these specimens was to permit using counts along random paths without mistaking concentration peaks from sub-surface inclusions with true manganese maxima.

Microradiographic techniques were used to determine the relative amount of porosity in various unidirectionally solidified castings. Specimens 1/2 x 1/2 x 0.0025 inches thick were radiographed using unfiltered copper and cobalt K-alpha radiation respectively. The two types of radiation were selected to distinguish between porosity and nonmetallic inclusions. The inclusions in this steel are mostly (FeMn)O·Al_2O_3, manganese sulfides and various oxides; they do not show up in radiographs using unfiltered cobalt radiation. The operating conditions were as follows: specimen and film to source distance 18 inches; for the copper target, the voltage was 35 KV, current 23mA, exposure time 17 sec; for the cobalt target the voltage was 45 KV, current 6mA, exposure time 25 sec. Contract Process Ortho negative film was employed which permitted good resolution of porosity up to a magnification of 50X. The amount of porosity was determined as the number of pores per unit area of specimen. Conversion to volume percent can be readily performed if the pores are essentially spherical. This was not the case and only a relative measure was employed. The resolution obtained with this procedure permitted detection of pores of 4 microns in diameter or larger.

Two .212" diameter tensile specimens were utilized: one had the gage length machined to a 2" radius with a 0.212" specimen diameter; the second had a constant diameter over the gage length. Ductility measurements were limited to reduction in area for radiused specimens. Both the reduction in area and elongation were determined for other specimens. The tensile properties of cylindrical castings were measured both normal and parallel to the chill, referred to as vertical and horizontal specimens respectively. The properties of keel blocks were measured with specimens whose axes were parallel to the long direction of the keel block leg.

The heat treatment conducted for the specimens after sectioning the casting was as follows: homogenizing for 2 hours at 2200°F, air cool; normalizing for 2 hours at 1750°F,

air cool; spheroidizing over night at 1150°F, air cool. The specimens were then rough machined and heated to 400°F, held for 9 hours and air cooled, followed by austenizing for 1 hour at 1650°F, cooling to 1500°F, holding for 1 hour and subsequent quench in oil, a double temper treatment of 2 hours at 400°F with a subsequent oil quench concluded the heat treatment.

RESULTS AND DISCUSSION

The detailed results and a discussion of these results are presented in the three interim technical reports that have been submitted on this contract. These reports are all concerned with the area of control of grain size and the relation between microstructure and mechanical properties in cast, high strength 4335 steel. In addition to the interim technical reports submitted, three publications in the technical literature have resulted from this contract. These are the following:

Wallace, J. F., Church, N., and Wieser, P., "Control of Cast Grain Size of Steel Castings," Modern Castings, April, 1966, p. 129, AFS Trans., Vol. 74, p.113.

Wallace, J. F. and Wieser, P. F., "Grain Refinement Cast Steel by Vacuum Melting," Cast Metals Research Journal, March, 1966, Vol. 2, No. 1, p. 1.

Wallace, J. F., Church, N, and Wieser, P., "Grain Refinement of Steel Castings," Journal of Metals, June, 1967, p. 44.

This latter paper was granted the outstanding paper award for 1966 by the AIME Electric Furnace Conference of the Iron and Steel Division. This paper will also appear in the 1966 Electric Furnace Proceedings of AIME.

GENERAL SUMMARY

This work is summarized by presenting the abstracts from each of the interim technical reports submitted on this material.

A. Report of September, 1964

Techniques for the quantative evaluation of refinement of a high strength cast steel have been evolved and used to test the effectiveness of various inoculants in achieving microstructural and macrostructural refinement. Of the various materials tested only titanium additions produced significant refinement of both the micro and macrostructure.

Comparison of the tensile properties of the basic steel with that of a refined steel shows that large additions (0.2-0.6 w/o) of titanium are sufficient to decrease the reduction in area sharply. Further data on castings partially refined with small (o.10 w/o) titanium additions indicate that an improvement in reduction of area is obtained compared to the results with a columnar structure solidified under similar thermal conditions.

B. Report of November, 1965

Inoculation techniques were developed to refine the as cast structure of high strength steel. Titanium was found to be the most effective inoculant. Large titanium additions (0.6%) form sulfides which embrittle the steel. Small titanium additions (0.1%) together with controlled superheat and mold design may produce significant refinement. Under these conditions the structure of unidirectionally solidified castings was changed from columnar to equiaxed and the reduction in area increased by approximately 25% for specimens oriented vertically to the direction of heat flow and increased about 75% for horizontally oriented specimens. A small improvement in reduction in area of keel block castings was obtained by this method. Some improvement in toughness was also obtained by this grain refinement. The influence of vacuum melting on the cast structure of the same steel was investigated. A hypothesis explaining the mechanism of refinement by vacuum melting was proposed.

C. Report of May, 1967

The relation of dendrite structure, nonmetallics and microporosity to solidification time and refinement by inoculation was investigated and correlated to mechanical properties of AISI 4335 steel castings.

The dendrite spacing is determined by solidification time and significantly influenced by solute content. Titanium and boron effectively refine the secondary dendrite spacing; selenium,

tellurium and sulfur have the opposite effect. Titanium inoculation also nucleates equiaxed dendrites, therby reducing or eliminating the columnar dendritic structure; the elements boron, selenium, tellurium, and sulfur may increase the length of columnar dendrites. This effect is hypothesized to result from entrapment and deactivation of nuclei in the segregates of these elements. The amount of nonmetallics or the solidification time are related to the ductility of unidirectionally solidified castings. Improvements in ductility from grain refinement can be expressed by the change in dendrite spacing. Refinement of the dendritic structure improves ductility, presumably by the reduction in particle size and the improved distribution of nonmetallics. Microprobe analysis indicates no significant differences in the segregation behavior of the elements manganese and chromium from inoculations. A hypothesis concerning the formation of dendrite arms was postulated based upon measurements of solute distribution and existing theories of dendrite arm formation.

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CONTROL OF CAST GRAIN SIZE OF STEEL CASTINGS EFFECT OF STRUCTURE AND NONMETALLICS ON PROPERTIES
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This project was concerned with determining the influence of microstructural refinement on the mechanical properties of cast high strength 4335 steel. It was determined that the addition of approximately 0.01% Ti to the steel subsequent to deoxidation provided considerable structural refinement. Such refinement was accompanied by improvements in the tensile ductility and impact resistance of the steel. This improvement in properties was particularly marked on specimens located with their long axis perpendicular to the direction of heat flow. Studies of the microstructure of the cast steel determined that nonmetallic inclusions were the primary factor (in the absence of microshrinkage) in determining the level of ductility. Improved ductility was also obtained with the final secondary dendrite arm spacing that is obtained by rapid solidification rates and by grain refinement with titanium.

1. Steel Castings
2. Grain Size Effects
3. Mechanical Properties

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